GEORGIA INSTITUTE OF TECHNOLOGY — OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

REVISED INFORMATION ONLY ☐ ORIGINAL ☒ REVISION NO. 1

Project No. ~~E-26-602~~ E-25-626 R5717-0A1 GTRC/~~GIT~~ DATE 3 / 14 / 85

Project Director: Dr. G. G. Eichholz School/~~Dept~~ ME

Sponsor E. I. Dupont de Nemours and Co., Aiken, S. C.

Type Agreement: C. O. AX-0630772-1 (DOE Prime-DE AC09-76SR00001)

Award Period: From _____ To 12/31/85 (Performance) 12/31/85 (Reports)

Sponsor Amount:	This Change	Total to Date
Estimated:	$	$
Funded:	$ 49,700	$ 99,482

Cost Sharing Amount: $ _____ Cost Sharing No: _____

Title: _____

ADMINISTRATIVE DATA OCA Contact Dennis Farmer x-4820

1) Sponsor Technical Contact:

DR. JOHN A. STONE - WASTE DISPOSAL TECH. DIV
E. I. DuPONT de NEMOURS & Co.
SAVANNAH RIVER PLANT
AIKEN, SC 29808-0001

2) Sponsor Admin/Contractual Matters:

Defense Priority Rating: _____ Military Security Classification: _____

(or) Company/Industrial Proprietary: N/A

RESTRICTIONS

See Attached _____ Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval – Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with _____

COMMENTS:

Change Order No. AX-0630772-1 adds $49,700 and extends the period of performance through 12/31/85.

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Project Director	Procurement/EES Supply Services	GTRC
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Accounting	Research Communications (2)	Other A. Jones

FORM OCA 85 PRE

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date 4/21/86

Project No. E-25-626 (Formerly E-26-602) School/~~CHE~~ ME (Formerly NE)

Includes Subproject No.(s) N/A

Project Director(s) G. C. Eichholz GTRC / ~~GIT~~

Sponsor E. I. DePont De Nemours & Co.

Title Environmental Impact of Buried Metallic Mercury

Effective Completion Date: 12/31/85 (Performance) _____ (Reports)

Grant/Contract Closeout Actions Remaining:

- [ ] None
- [XXX] Final Invoice or Final Fiscal Report
- [XXX] Closing Documents
- [XXX] Final Report of Inventions Questionnaire sent to P.I.
- [XXX] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other _____

Continues Project No. _____ Continued by Project No. _____

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A. Jones
R. Embry

FORM OCA 69.285

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March 11, 1985

Please reply to:
NUCLEAR ENGINEERING AND
HEALTH PHYSICS PROGRAM
CHERRY EMERSON BUILDING
GEORGIA INST. OF TECH.
ATLANTA, GEORGIA 30332 U.S.A.

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Co.
Aiken, SC 29808

E 25-626

Monthly Progress Report - Project E-25-626

Dear Dr. Stone:

Since I reviewed the status of this project with you at SRP at the end of February, there is little to add. We are in the process of running the cold-vapor system on a series of samples in the ppb range and are making up a number of mercury - contacted water samples to obtain data on diffusion/solution contamination levels.

We are obtaining data on low-level mercury tracers, to see if the specific activity of our present Hg-203 material is detectable at very low concentrations. If not, we will have to prepare a higher-activity tracer material.

During the next few weeks we expect to set up some low-level adsorption tests in stoppered flasks and some column tests using GT sand and SRP soil.

Please call me if you have any questions.

Yours sincerely

G.G. Eichholz
Regents' Professor

GGE/swm

cc: Dr. J.L. Carden
P. Heitmuller

Telephone: 404-894-3720 Telex: 542507 GTRIOCAATL Fax: 404-894-3120 (Verify: 404-894-4850)
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April 10, 1985

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ATLANTA, GEORGIA 30332 U.S.A

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E. I. Du Pont de Nemours & Co.
Aiken, SC 29808

Monthly Progress Report - Project E-25-626

Dear Dr. Stone:

Progress was slowed during the past month when then cold-vapor cell was contaminated as a result of some solution samples that contained relatively high mercury concentrations in suspension. The cell has since been cleaned up and the system is back in service.

A series of low-concentration samples have been prepared and are being analysed to follow up on your suggestions to start at the low end of concentration ranges to observe if any surface attachment occurs.

Mr. Frank Petelka has joined this project and has set up several soil columns with embedded mercury droplets to measure the solution or entrainment, if any, in a slowly flowing stream. A number of samples have been collected and are awaiting analysis.

Some problems with our counting system have delayed assessment of the limit of detectability of our activated mercury in dilute concentration, and we expect to check this next week. A freshly activated sample will be irradiated, if necessary.

Please let me know if you have any questions.

Yours sincerely

G.G. Eichholz
Regents' Professor

GGE/swm

cc: Dr. J. L. Carden
P. Heitmuller (OCA)

Georgia Institute of Technology
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June 7, 1985

Please reply to:
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CHERRY EMERSON BUILDING
GEORGIA INST. OF TECH.
ATLANTA, GEORGIA 30332 U.S

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Co.
Aiken, SC 29808

Monthly Progress Report - Project E-25-626

Dear Dr. Stone:

During your visit here on May 22, we reviewed the present status of this project with you and Dr. Oblath. The following is a brief summary.

A. Analytical work
The atomic absorption spectrometer has attained a lower limit of detection of 0.5 ppb. The radioactive tracer tests seem to be able to go down to about 20 ppb. We plan to check the overlap region, below 100 ppb. for consistency. Problems are expected to arise for colloidal and suspended material that can only be handled by the tracer method.

B. Coupon absorption test
Tracer tests are continuing with samples that are slowly rotating in a beaker with active mercury at the bottom. The introduction of a galvanized sample led to a dramatic transformation and a film formed, presumably of tin, on the mercury droplet. The test will be repeated and quantified for fresh samples, without mixing them in the same run.

C. Static test
We have taken pictures of the static, Hg-covered soil columns and are now getting ready to dissect them.

D. Flow columns
The flow tests with equilibrated water flowing around an Hg droplet embedded in soil columns are continuing and will be run over longer periods. We are planning to supplement them with flow tests involving kaolin suspensions and water containing kerosene or chelates.

We will continue to stay in touch and expect to submit the results of the coupons tests to you with our next monthly report.

Yours sincerely

G.G. Eichholz
Regents Professor

GGE/swm

cc: P. Heitmuller (OCA)

E-25-

Georgia Institute of Technology

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SCHOOL OF MECHANICAL ENGINEERING

July 10, 1985

Please reply to:

NUCLEAR ENGINEERING AND
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CHERRY EMERSON BUILDING
GEORGIA INST. OF TECH.
ATLANTA, GEORGIA 30332 U.S.A.

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, SC 29808

Monthly Progress Report - Project E-25-626

Dear Dr. Stone:

During the past month, work has progressed along the lines indicated in our last monthly report. Mr. Harold Haynes has joined the project, replacing Matthew McFee, and is trying out methods for dissecting the static seepage columns.

Following your previous request we are attaching more detailed reports to outline the results obtained for two of the experiments; the adsorption measurement on coupons of potential lysimeter liners, and column tests to measure mercury entrainment. The coupon tests show that mercury attachment is significantly higher on galvanized iron; the figures obtained are clearly too low as they do not take into account a visible scale that tended to flake off.

The column test show that there clearly is some movement of mercury and future tests will have to be done to establish whether this is due to physical entrainment, expansion of the column material or adsorption on suspended particulates. All of these tests are being continued.

Please call me if you have any questions.

Yours truly,

G. G. Eichholz
Regents' Professor

GGE/jg

cc: P. Heitmuller

Interim Progress Report

Mercury Project (E25-626)

M. F. Petelka

July 9, 1985

Coupon Tests

In an effort to determine the extent of the interaction between dissolved mercury in water and several possible lysimeter construction materials, a test was set up in which small (typically 25mm X 35mm) coupons of various construction materials were placed in a beaker(containing dilute mercury.) Three materials were chosen for the initial test; aluminum, fiberglass, and galvanized iron; all samples were run in duplicate. To remove surface contaminants that may effect mercury adsorption, the coupons were washed in a detergent solution, rinsed afterwards in deionized water and then acetone, and allowed to air-dry. The edges of the fiberglass coupons were dipped in melted paraffin to seal the ragged edges resulting fr the cutting of the coupons from a larger sheet. It was felt that the edges could adsorb water by capillary forces, increasing the mercury concentration preferentially compared with the relatively nonporous surfaces.

The dilute mercury solution was produced by placing a small drop of activated (Hg-203) mercury at the bottom of a beaker containing de-ionized water. After an extended period of standing the water was found to contain 60 ppb of mercury. This solution was then used to measure mercury sorption on the sample coupons by counting the ^{203}Hg activity in the surface layer.

The coupons were suspended in a beaker containing 500 ml of deionized water in equilibrium with activated metallic mercury. The stirring apparatus was used to rotate the coupons slowly (approximately 10 rpm) to minimize concentration variations in the solution. The coupons were immersed for 10 days, at which time they were removed from solution and allowed to air dry. Photographs were taken of the coupons (Figure 1). The galvanized coupons reacted dramatically, becoming textured with a white substance while releasing a large volume of white powder that settled on the bottom of the beaker as well as attaching to the surfaces of the other coupons. Figure 2 shows the accumulation, at 1.8x magnification. This white powder was shown to contain 0.03 wt% mercury.

To calibrate the system a standard coupon was fabricated by evaporating 1.09 g of water containing 60 ppb Hg uniformly on the surfaces of a copper coupon. This standard coupon was counted in the same manner as the test coupon and a count rate to amount-of-mercury realtionship was established

Table 1 shows the results of the mercury analysis of the coupons. After counting, the coupons were rinsed with deionized water to remove the white powder and to estimate the bonding strength of the mercury. The aluminum and fiberglass coupons showed no significant reduction in activity upon washing. The galvanized coupon showed a 26.1% reduction in attached mercury.

Table 1

Concentration of Mercury on Coupon Surfaces

Coupon material	Amount of Mercury (g/cm^2)
Aluminum	6.1×10^{-8}
Fiberglass	5.9×10^{-8}
Galvanized	3.0×10^{-7}
Galvanized, rinsed	2.0×10^{-7}

Preliminary information shows that of the three materials chosen for this first test, galvanized iron is not an acceptable choice due to structural degradatio

An experiment was run to determine the kinetics of the adsorption of mercury on the aluminum and fiberglass coupons. Fiberglass coupons and aluminum coupons were immersed in mercury-equilibrated water as in the first experiment. Coupons were removed after 24, 48, and 72 hours of contact, rinsed, and counted (Table 2).

The coupons reached values comparable to the first 10-day test within the first 24 hours of contact. Greater variation is to be expected due to the fact that this test was conducted with individual coupons at each immersion time, while the other coupon tests were run in replicate.

Table 2

Coupon material	Time of contact	Amount of mercury adsorbed (g/cm^2)
Fiberglass	24 hours	5.1×10^{-8}
Fiberglass	48 hours	4.7×10^{-8}
Fiberglass	72 hours	8.4×10^{-8}
Aluminum	24 hours	1.2×10^{-7}

Another coupon test is underway to determine the effect of mercury concentratio on adsorption. Coupons have been immersed in a solution containing 30 ppb mercury i deionized water, one half the concentration used in the earlier tests. Results are expected soon.

Column Flow Tests

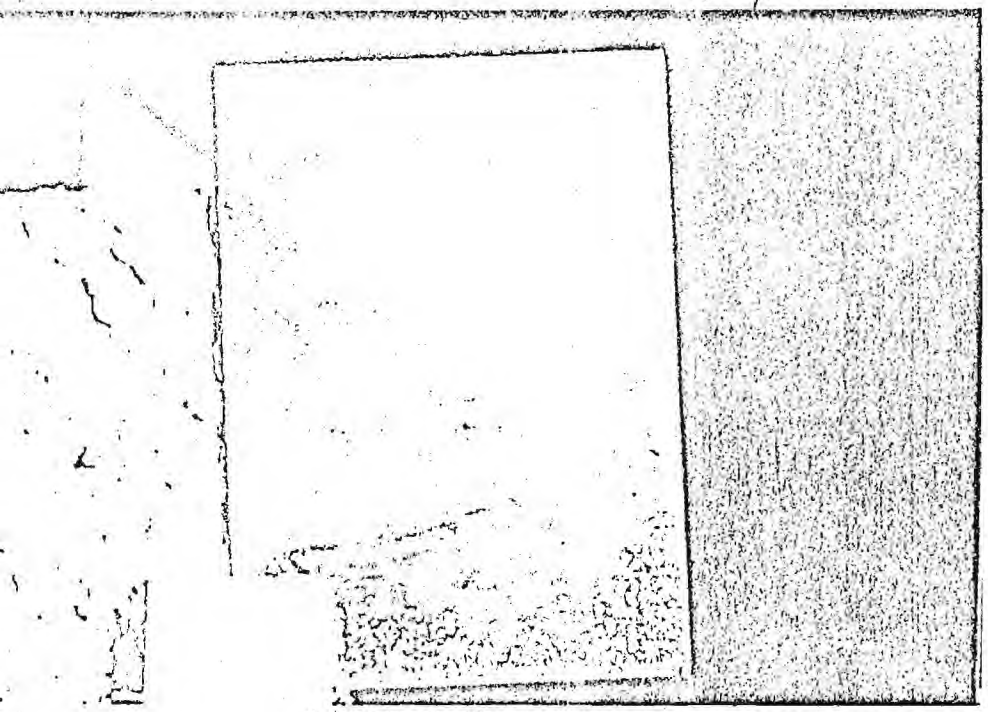
A 1 1/2 inch ID plastic column was packed with GT Sand to a height of 18 cm and placed in water to saturate. The column volume was 205 cm^3, the pore volume 98.5 cm^3. The column was placed in a system to allow saturated flow, with recycling, at a controlled flow rate.0.076 g of activated mercury was placed on the soil surface (6.6 mg/cm^3).

The flow rate of water through the column was controlled by a valve located at the bottom of the column. An overflow line was attached to the column above the soil level to insure continued saturation. The water was pumped from a reservoir to the top of the column. The column effluent was collected in a graduated cylinder to verify the cumulative water flow. Samples were periodically collected from the effluent and analyzed for mercury concentration. The volume of water collected in t graduated cylinder was recorded and the water was returned to the reservoir to be recycled.

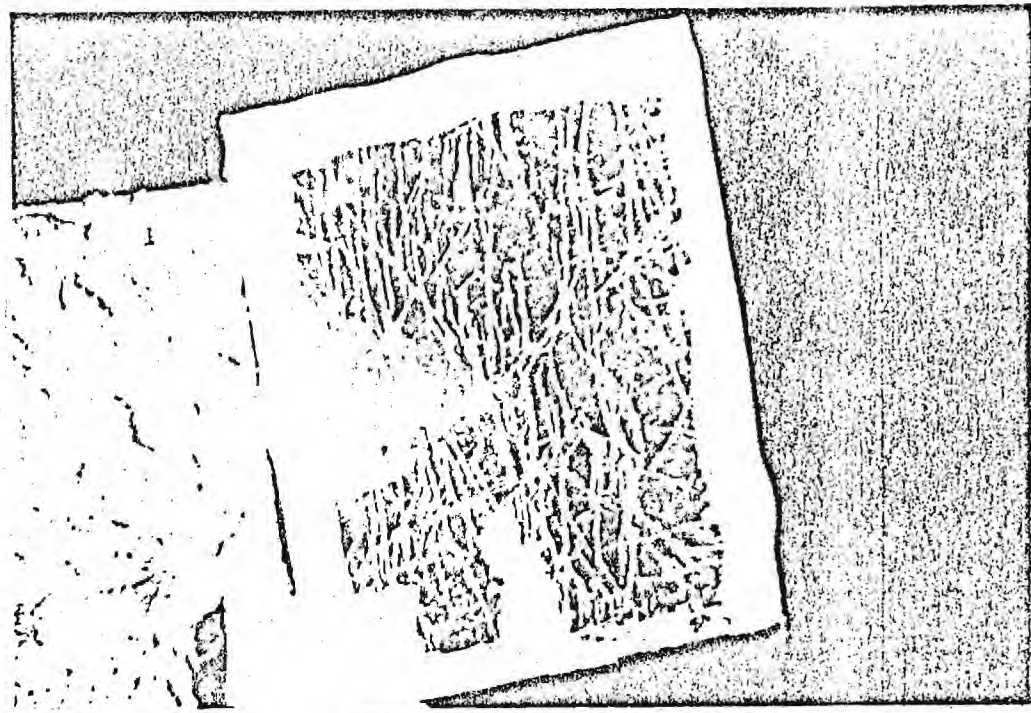
The samples were analyzed for mercury concentration using a 5-inch sodium iodid well detector housed in a 6 inch iron shield, coupled to a single channel analyzer set to accept the 0.279 MeV photopeak from Hg-203. The MDA of this system for mercu in water is 20 ppb, the Hg concentration in water is approximately 60 ppb.

Effluent samples were taken at 1,2,4,8,16,30,40,50, and 60 pore volumes and analyzed for mercury activity. The activity in all cases was below 20 ppb. The test was stopped after 60 pore volumes (6 liter) due to equipment malfunction. It was felt that the reliability of the data produced after restart did not warrant extended testing. The samples are being analyzed using Atomic Absorption to verify the results obtained from the sodium iodide system.

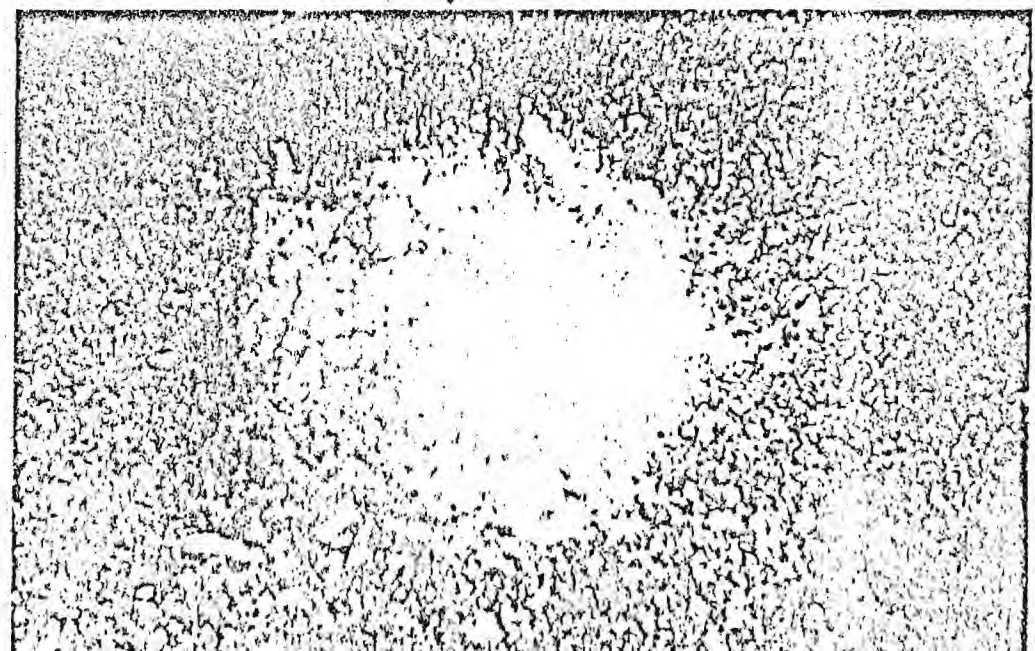
coupon Test materials



ALUMINUM



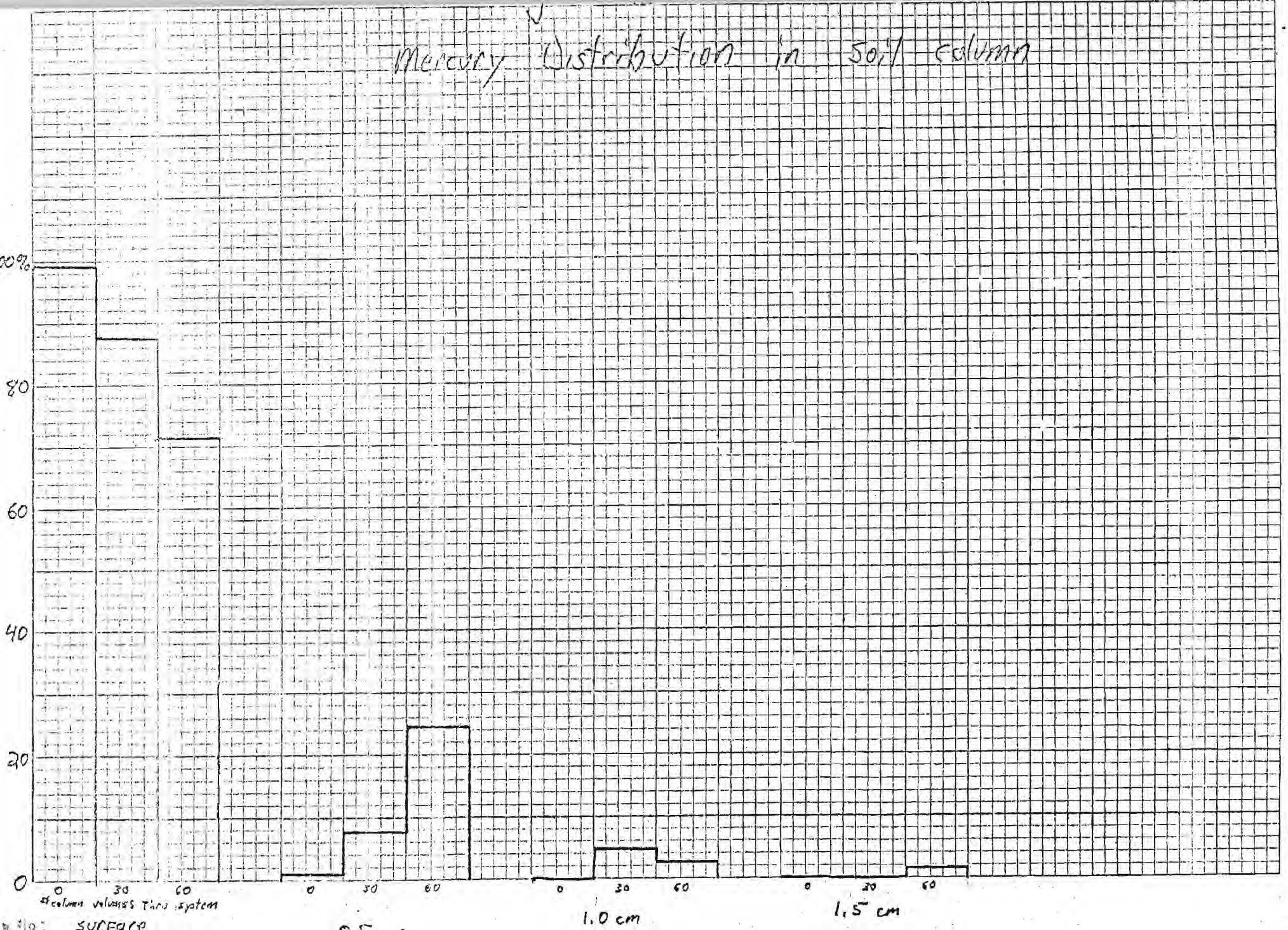
Fiberglass



Vertical Distribution in the Flow Column

A sodium iodide crystal was used in conjunction with an MCA to determine the distribution of the activated mercury as a function of time and horizontal location. The detector was placed inside a lead collimator to allow high resolution during the vertical scans. The collimator has a horizontal window 3 mm high by 45 mm deep. The window gives a total scanning height at the column center of 5.6 mm. Measurements were taken at 0.5 cm intervals down the column starting at the surface. Scans were taken at three times; before start of water flow, after 30 column volumes had passed through the system, and after 60 column volumes had passed. 30 column volumes represents approximately 263 cm of infiltrating water, 60 volumes is approximately 526 cm, or 207 inches. Figure 3 shows the distribution of Hg activity with depth for the three scans. The mercury is seen to be moving down the column with the flow of water. After 60 column volumes (approximately 6 liters) have moved through the system, the activity at the 0.5 cm level is 24% of the activity at the column surface. The actual concentration of mercury in each horizon has not been determined; the column will be sectioned at a later time.

Additional tests are needed to identify the nature of the migration mechanism and the retention mechanism.


Mercury Distribution in Soil Column
100%
80
60
40
20
0
0
30
60
0
30
60
0
30
60
0
30
60
1.0 cm
1.5 cm

E-25-626

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ATLANTA, GEORGIA 30332 U.S.A.

August 9, 1985

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E. I. Du Pont de Nemours & Company
Aiken, SC 29808

Monthly Progress Report-Project E-25-626

Dear Dr. Stone:

During the past month work has continued in two areas:

1. Adsorption on potential construction materials.

Duplicate coupons of aluminum, fiberglass and galvanized iron were immersed in 30 ppb activated mercury solutions, roughly half the previous concentration. Adsorption on the coupons was counted after 3.5 days immersion and yielded comparable attachment, $5\text{-}7\text{-}10^{-8}$ g Hg per cm^2 for all the materials, with somewhat less white powder formed in the case of the galvanized sample. To measure the rate of attachment, duplicate fiberglass and aluminum samples were exposed in 60 ppb deionized water for 1, 2, 3, and 10 days. Adsorption on fiberglass seemed to level off within the first day, while that on aluminum seemed to take place more slowly.

2. Column seepage tests

A test column has been set up to try out a procedure for extruding the seepage columns from their plastic tubing without cross contamination. A pressure jack system has been worked out. Analyses have been done on removed soil and have shown no mercury transfer. The standing columns will be extended, dissected and analyzed in the next two weeks.

Some problems were encountered with fluctuations in the atomic absorption systems, but they seem to have been overcome. We expect to start on dissolution tests in various aqueous media in the immediate future. Please call me if you have any questions regarding this work.

I expect to be at SRP August 20-21 in connection with the RSAC meeting and hope to have a chance to meet with you to settle any questions regarding our proposal for further work on soil hydraulics and model validation.

Yours truly,

Geoffrey G. Eichholz
Regents' Professor

GGE:jp

cc: P. Heitmuller

E-25-626

Georgia Institute of Technology

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September 17, 1985

Please reply to:

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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, S.C. 29808

Monthly Progress Report-Project E-25-626

Dear Dr. Stone:

Though work progressed at a slightly reduced rate during exam periods and the quarter break some momentum was maintained, nevertheless, in the two areas of activity.

In the mercury solubility work, active mercury was maintained in contact with various water mixtures, which were stirred gently. After appropriate times an aliquot was counted and another retained for AA analysis. The solutions involve 10%, 1% and 1 ppm kerosene in deionized water and 1% and 5ppm EDTA in water respectively. The results are summarized on the attached sheet and indicate, not unexpectedly, that high concentrations of EDTA or kerosene will mobilize mercury.

A fair amount of effort has been devoted to the dissection of the static soil seepage columns. This has required a disproportionate effort to avoid cross contamination of layers by mercury attachment to the tube walls. The procedure evolved consists of reverse ejection of the drained columns, bottom first, and removal of thin slices as they emerge for analysis. To prepare the samples for AA analysis, each soil slice has to be treated to solubilize any contained mercury. This work continues and we hope to conclude it shortly.

Please call me if there are any questions regarding this work.

Yours sincerely

G.G. Eichholz
Regents' Professor

cc: P. Heitmuller (OCA)

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Mercury Project

The following solutions were made up and allowed to mix gently. After mixing, a 1 ml sample was counted.

#	Sample	Count rate/hr ± σ
1	10% Kerosene/DI water	598 ± 132 , 366 ± 132
2	10% Kerosene/DI water	265 ± 101
1	1% Kerosene/DI water	488 ± 99.9
2	1% Kerosene/DI water	below background
1	10^{-6} Kerosen-/DI water	"
2	10^{-6} Kerosene/DI water	"
1	1% EDTA / DI water	6579 ± 116
2	1% EDTA/DI water	4430 ± 107
1	5×10^{-6} EDTA/DI water	background
2	5×10^{-6} EDTA/DI water	"

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October 9, 1985

Please reply to:
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Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E.I. Du Pont de Nemours & Company
Aiken, S.C. 29808

Monthly Progress Report-Project E-25-626

Dear Dr. Stone:

We are making a concentrated effort to complete our measurement program. The soil columns have been dissected and samples prepared for analysis. Some stability problems with the atomic absorption equipment and limitations in access are slowing down the rate of analyses, but we expect to overcome this problem by some additional effort.

The active mercury has decayed to a level where counting statistics are undesirably low and we are in the process of reactivating a mercury sample in the GTRR reactor. This should be available this week, but the sample has to undergo a cooling period for the decay of short-lived isotopes.

Additional data are being obtained on mercury transfer from dilute solution onto structural coupons in the presence of kerosene, EDTA and dodecane and on the uptake on suspended particulates under those conditions. It is expected that better results will be obtained, relatively quickly, with higher specific activity mercury tracer.

We are also setting up a soil column for additional measurements on the migration of dissolved mercury in dilute concentrations, though we do not expect to observe significant differences from the water movement itself. Please call if you have any questions regarding this work.

Yours sincerely

G.G. Eichholz
Regents' Professor

GGE/sm
cc: P. Heitmuller (OCA)

Mercury Project Update 10-16-1985 M. Frank Petelka

Solubility: The effects of complexing agents were investigated using aqueous solutions containing $5X10^{-6}M$ Kerosene and $1X10^{-2}M$ and $5X10^{-6}M$ EDTA. Metallic mercury was added and samples were agitated gently to allow the samples to equilibrate. An aliquot of each sample was then taken and analyzed for mercury concentration using a sodium iodide detection system. Duplicate samples were analyzed using a cold-vapor atomic absorption system.

RESULTS

Sample	Mercury Concentration (ppm)	
	Counting system	Atomic Absorbance
$1X10^{-6}M$ Kerosene	2.16 +/- 0.51	0.799
$1X10^{-2}M$ EDTA	83.30 +/- 1.35	83.616
$5X10^{-6}M$ EDTA	1.51 +/- 0.52	1.386
Deionized Water	4.83 +/- 0.62	4.717

The results indicate a good agreement between the two systems. The counting system results of the kerosene test were verified by repeating the analysis. The limit of detectability for the sodium iodide system is 0.42 ppm. The mercury is being reactivated to reduce the LLD. The next round of samples will include dodecane, EDTA, and water.

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ATLANTA, GEORGIA 30332 U.S.A.

December 9, 1985

Dr. John A. Stone
Waste Disposal Technology Division
Savannah River Laboratory
E. I. Du Pont de Nemours & Co.
Aiken, S. C. 29808

Monthly Progress Report - Project E25-626

Dear Dr. Stone:

We are now reaching the concluding phase of this project and this will be the final monthly report letter. We are in the process of assembling the Final Report and expect to have it all prepared by Christmas. A few final counts and analytical results are still coming in.

The project has benefited substantially from the availability of some extra manpower this quarter under our graduate research development program ("Stelson Plan"). I have discussed the general status and the results obtained at a meeting with Dr. Oblath at SRP on November 21, 1985.

Please call me if there are any questions regarding the wind-up of this project.

Yours sincerely,

Geoffrey G. Eichholz
Regents' Professor

GGE:jp

cc: P. Heitmuller (OCA)

Telephone: 404-894-3720 Telex: 542507 GTRIOCAATL Fax: 404-894-3120 (Verify: 404-894-4850
AN EQUAL EDUCATION AND EMPLOYMENT OPPORTUNITY INSTITUTION

ENVIRONMENTAL IMPACT OF BURIED METALLIC MERCURY:

Phase II

Final Report

Project E25-626

SRP Purchase Order AX-0630772-1

Geoffrey G. Eichholz

Project Director

Submitted to

Waste Disposal Technology Division

Savannah River Laboratory

E I Du Pont de Nemours & Co.

Aiken, S. C. 29808

Nuclear Engineering and Health Physics Program,

George W. Woodruff School of Mechanical Engineering,

Georgia Institute of Technology

Atlanta, Georgia 30332

December 1985

CONTENTS

LIST OF FIGURES

SUMMARY

The work described in this report continued an investigation undertaken in support of studies conducted by the Savannah River Laboratory to delineate any risks that may be associated with the known presence of metallic mercury in some on-site disposal trenches. The objective of this phase of the investigation was to study possible entrainment of mercury in ground water and contaminated trench fluids, to determine the parameters needed in planning a possible lysimeter project on this problem, and to simulate the behavior of metallic mercury in soil columns under static and dynamic conditions.

The solubility and uptake of mercury in water and soil columns was monitored by means of atomic absorption spectrometry and by radioactive tracer methods. Static tests on wet and dry soil columns exhibited limited penetration of a few millimeters, presumably under the static pressure head but little further migration. Migration in flow columns was negligible, except in the presence of EDTA chelant. No migration by means of attachment on suspended particulates was observed.

In a separate series of tests on potential construction materials for lysimeters silos previous results were confirmed that rapid mercury transfer occurs to galvanized iron, leading to the formation of visible scale, but no appreciable adsorption on any of the other materials tested.

In general, mercury migration from the disposal trenches would be expected to occur by slow dissolution in contacting water to maximum concentrations of the order of less than 10 parts per billion. This would presumably a very minor addition to the general level of contamination of trench drainage.

PROJECT PERSONNEL

(all part-time)

Geoffrey G. Eichholz, Ph.D	Project Director
M. Frank Petelka, M.S. H.P.	Senior Graduate Research Asst.
Iwona S. Miazek, M.S.	Graduate Research Asst.
Matthew F. McFee, B.S. H.P	Graduate Research Asst.
P. Tennent Slack, B.S.	Graduate Research Asst.
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INTRODUCTION

Environmental pollution by mercury has always been recognized as having potentially serious effects, but little attention was paid to it until the appearance of detectable amounts of mercury in fish caught in Sweden and the Great Lakes in the late 1960's and the diagnosis of Minamata disease in Japan and other countries as a result of waste mercury dumping in ocean bays and its use in various fungicides. A substantial investigation was started at Oak Ridge National Laboratory in the past four years in response to the discovery of mercury in the Clinch River, that was traced to effluent disposal from the Y-12 Plant there.

Since it was known that some metallic mercury was buried at several locations at the Savannah River Plant, Orebaugh and Hale, in 1976 (1), conducted an extensive study on mercury transport, primarily by gaseous diffusion of mercury vapor to the atmosphere. They estimated that mercury from this source, even under worst-case conditions, would contribute only amounts in the range of natural concentrations to local surface streams. To supplement that work, the present project was initiated in 1983 to provide information on the pathways and potential concentrations of mercury migration from the disposal areas into the groundwater system.

The first phase of this project was reported a year ago (2). It had as its main objectives (a) the compilation of a critical review of the literature; (b) the establishment of analytical facilities for trace concentrations of mercury in water; and, (c) the preliminary measurement of mercury uptake in water and of the adsorption of mercury on potential structural materials for lysimeter studies.

The first two of those objectives were completed. The literature review was completed and submitted to SRL as a topical report (3). A cold

vapor cell was constructed for atomic absorption spectrometry and the system was calibrated, as reported previously (2). The other measurements formed the basis for the subsequent work described in this report.

The objectives of the present project may then be described as follows:

1. To measure the solubility and entrainment of metallic mercury into standing or moving water in contact with the metal;

2. To provide data necessary for a realistic assessment of the potential environmental impact of buried mercury under site-specific conditions at SRP; and

3. To supply adsorption coefficients for dilute dissolved mercury on structural materials that may be employed for further investigation of this matter.

In addition, the literature in the field continued to be monitored with only few recent additions to the bibliography. Relevant publications are listed in the References (4-9). The most significant addition to the literature is the report by Rothschild et al. on the Oak Ridge investigation on subsurface mercury (4).

DETAILS OF TEST WORK

1. Analytical Procedures

A. Atomic Absorption

Since the mercury concentrations in water and adsorbed on surfaces were expected to be very low, atomic absorption spectrometry was employed to measure dissolved mercury in the parts-per-billion (ppb) range and radioactive tracer techniques at higher concentrations and for non-aqueous samples. Samples were prepared in the overlap region, around 60 ppb, to verify agreement and consistency between the two methods.

The atomic absorption method itself was based on the use of a cold-vapor cell apparatus which was described in detail in the previous report (2). Details on the setting-up and operation of the system are provided in Appendix A for the record.

The system performed quite well and appeared to give consistent results. Care had to be taken not to contaminate the cell and samples had to be selected in order of increasing Hg content to avoid repetition of one incident that took the system out of use for an appreciable period for cleanup. Lower limit of detection was established previously at about 1 ppb.

B. Radioactive Tracers

Triply distilled mercury was activated in Summer 1984 in the Georgia Tech Research Reactor to provide about 1 ml of active mercury. A smaller sample was irradiated first to confirm the absence of any interfering activity. On cooling, the main sample showed predominantly Hg-203 and Hg-197m activity, as expected. This material was used for all tracer tests for the early months of the present project. After that time the specific

activity of the material became too low to be useful and it was decided to reactivate the remaining mercury.

The second activation took place on Sept. 27, 1985. The tracer material, 1 g of liquid mercury, was put into use after a two-week cooling period, to allow decay of the 64-hour mercury-197 isotope.

Activation calculations and subsequent intercomparison of a highly diluted Hg tracer sample with the atomic absorption method showed that the active mercury had a specific activity of about 24mCi/g on Oct. 4, 1985. In practice, this called for appreciable dilution for actual tracer tests, which typically were run at the microcurie level.

Most samples were counted with a heavily shielded 5-inch scintillation detector, shown in Figure 1. At the operating conditions selected, background was of the order of 12,000 counts per hour. This relatively high background setting was chosen for good counting statistics as many of the samples were expected to have count rates at or near background.

2. Solubility Tests

The most basic process through which elemental mercury could enter subsurface water from a burial location would be by a dissolution or suspension process. Elemental mercury is listed as "insoluble" in both hot and cold water in the CRC "Handbook of Chemistry and Physics" (10). Waldbott (11) lists Hg concentration in ocean water with a range of 0.03-5 ppb, and in waters near mercury deposits as 5-100 ppb. A commonly accepted solubility limit for elemental mercury appears to be of the order of 70 ppb.

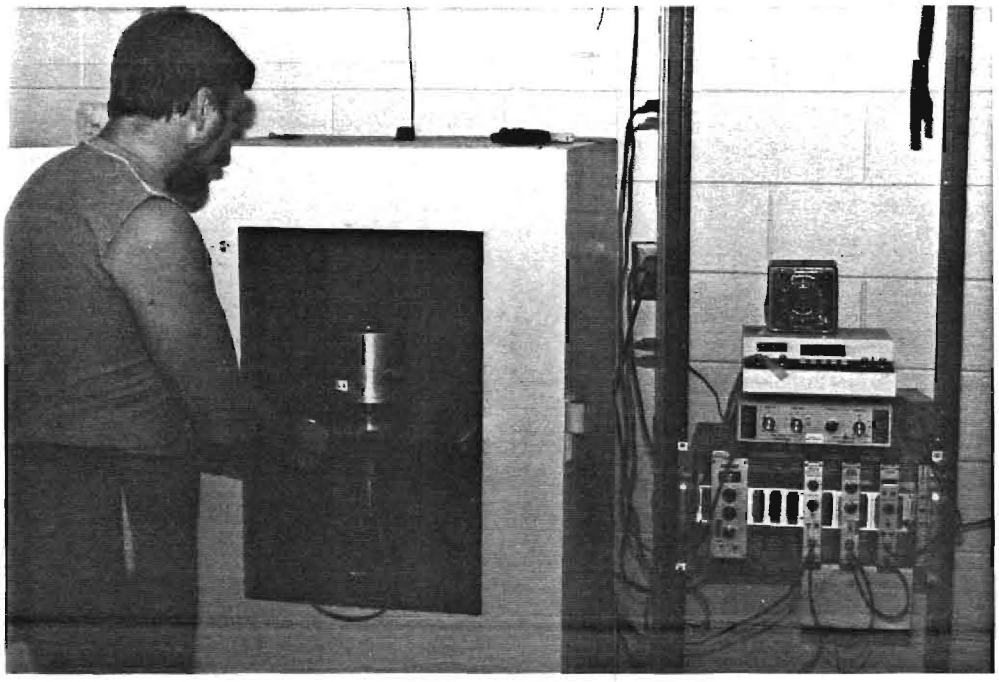


Figure 1. View of scintillation detector system

The degree of dissolution in trench water, presumably under anoxic conditions, is complicated by the uncertain quality and composition of the water involved and, for that matter, the purity of the mercury. In the case of the SRP trenches an additional complication arises, because of the possible presence of solvent extraction wastes containing organic solvents and, perhaps, some chelating reagents.

For this reason a number of tests were conducted to measure the uptake of mercury into solution for deionized water, soil-equilibrated water, and solutions containing commercial kerosene, dodecane and ethylene diamine tetraacetic acid (EDTA). A small drop of radioactive mercury was deposited at the bottom of a test tube and covered with the aqueous solvent. The test tube was then shaken gently for prolonged periods and the supernatant solvent was sampled and analyzed, both by counting and atomic absorption. All samples were run in duplicates and split for comparative analysis. Errors quoted are for one standard deviation.

A separate series of tests were conducted to measure the adsorption of dissolved mercury on suspended kaolin particles, using an aged suspension that had been sitting in carboys for over two years.

Test Details

Description of Method

40 ml of each solution was placed in 70 ml test tubes. A drop of activated Hg (typically 0.4 g) was placed at the bottom of the test tube. The tubes were stoppered and placed on a horizontal agitator (~2 Hz) for five days under gentle agitation.

10 ml of solution was withdrawn, to which 0.77 ml of preservative was added to minimize Hg losses. Aliquots were counted in the shielded sodium iodide well counter. Analysis was also performed using cold-vapor atomic

absorbance. All glassware was acid washed before use. Eppendorf pipets were used; the tips were discarded after every sample to minimize cross contamination. The samples were preserved by adding $K_2Cr_2O_7$ and HNO_3 to make: 5% (v/v) HNO_3 and 0.01% (w/v) (Cr_2O_7 (Fe 74).

Solutions:

Equilibrated GT Sand/H_2O:

Contact DI water with GT sand for 4 days. Filter water through 0.22 μ m millipore filter paper to remove solids.

Dodecane (MW = 170.34 g/mole; ρ = 0.749 g/ml):

Attempts to make aqueous solutions containing dodecane were unsuccessful. The organic formed a layer. Straight dodecane was used in tests.

10^{-2}M EDTA:

2.923 g of EDTA was dissolved in 1 L of deionized water.

10^{-4}M EDTA:

10 ml of the 10^{-2}M EDTA solution was diluted to 1 L using DI water.

10^{-6}M EDTA:

10 ml of the 10^{-4}M EDTA solution was diluted to 1 L using DI water.

Preservative:

0.13 g of $K_2Cr_2O_7$ was dissolved in 100 ml of 70% nitric acid.

Table 1 presents the results of background runs. It was found that the system was quite stable over several weeks.

Table 2 shows the results of the first tracer run on mercury uptake in several aqueous solutions.

Table 3 records the comparison between the AA analysis, on highly diluted samples, and the radioactive tracers.

TABLE 1

Counting System Background

Sample	Counts	Time (hr.)	$\sqrt{n}$	% error	CPH $\pm 1\sigma$
B_1	31678	2.52	178	0.56	12570 $\pm$ 71
B_2	155495	12.72	344	.25	12228 $\pm$ 31
B_3	163117	13.37	404	.25	12171 $\pm$ 30
B_4	157618	12.95	397	.25	12171 $\pm$30
B_5	107903	8.73	328	.30	12355 $\pm$38
B_6	136985	11.17	370	.27	12267 +33
B_7	169692	13.88	412	.24	12222 $\pm$30
B_8	162534	13.25	403	.25	12267 $\pm$30

Results of first run

TABLE 2

Activity per ml solution

#	SAMPLE	Countrate/hr $\pm$ σ
1	10% kerosene/DI Water	598$\pm$132, 366 $\pm$ 132
2	10% kerosene/DI water	265 $\pm$ 101
1	1% kerosene/DI water	488 $\pm$ 99.9
2	1% kerosene/DI water	below background
1	10^{-6} kerosene/DI water	below background
2	10^{-6} kerosene/DI water	below background
1	1% EDTA/DI water	6579 $\pm$ 116
2	1% EDTA/DI water	4430 $\pm$ 107
1	5×10^{-6} EDTA/DI water	background
2	5×10^{-6} EDTA/DI water	background

TABLE 3

Comparison of AA and Counting Results

Mercury Concentration (ppm)

Sample	Counting System	Atomic Absorbance
$1x10^{-6}$M Kerosene	2.16 ± 0.51	0.799
$1x10^{-2}$M EDTA	83.30 ± 1.35	83.616
$5x10^{-6}$M EDTA	1.51 ± 0.52	1.386
Deionized Water	4.83 ± 0.62	4.717

The results indicated a good agreement between the two systems. The counting system results of the kerosene test were verified by repeating the analysis. The limit of detectability for the sodium iodide system was 0.42 ppm for the mercury tracer used at that time.

Results of second run (with freshly activated mercury).

TABLE 4

Comparative results of second run

Sample	Net Count Rate (cph)	Atomic Absorption (ppb) 11-11	12-5	Aver.
Deionized water	107 ± 69	21.6	69.0 ± 4.8	45.3
Equilibrated water	71 ± 69	36.7	79.7 ± 10.3	58.2
EDTA 10^{-6}M	410 ± 74	70.5	53.6 ± 1.6	62.1
10^{-4}M	1184 ± 82	6100	7371 ± 1140	6736
10^{-2}M	9631 ± 106	27906	23039 ± 353	25473
Dodecane	478 ± 71	-	-	-

The dodecane sample was not amenable to AA analysis. The results all show very low mercury uptake and for the counting results only the higher EDTA concentrations can be considered to be significantly different from the background of about 12,000 cph. Overall the AA and counting results are in fair agreement and show that the mercury uptake in deionized and equilibrated water was at concentrations approaching the solubility limit. No appreciable complexing or dissolution appeared to occur in dodecane. Complexing of mercury seemed to take place for the higher concentrations of EDTA, certainly into the parts per million range. At these concentrations the upper limit of sensitivity of the AA system is reached and analysis

requires substantial dilution of the samples; the radioactive tracer results may represent a more reliable indicator.

The AA should provide the more reliable data at the low concentrations. The AA may underestimate the concentration of mercury contained in the EDTA solutions. The $SnCl_2$ may not completely reduce the mercury that is complexed. The cold-vapor technique relies on the reduction of mercury to the metallic state which diffuses from the solution into the carrier gas and is carried into the absorption cell.

3. Kaolin suspension test

The attachment of Hg to kaolin was studied by contacting the two, then removing the clay particles by centrifugation. The mercury concentration of each solution was determined by counting. The kaolin samples could not be run through the AA, owing to the possibility of the kaolin particles clogging the porous glass frit of the reducing vessel. The kaolin was contacted with the mercury for five days. Aliquots were removed for counting. Others were removed, centrifuged for 30 min. at full speed in our "international clinical centrifuge" to remove suspended material, and the supernate was counted. This suspension contains 9.6×10^{-5} g kaolin per ml H_2O.

The kaolin suspension counted 348 $\pm$ 132 cph.

The supernate counted 255 $\pm$ 148 cph.

These results indicate that the mercury is sorbed onto the clay particles and is present at a concentration higher than that in either DI or equilibrated water. The difference between the concentration in the supernate and the full suspension is not statistically significant.

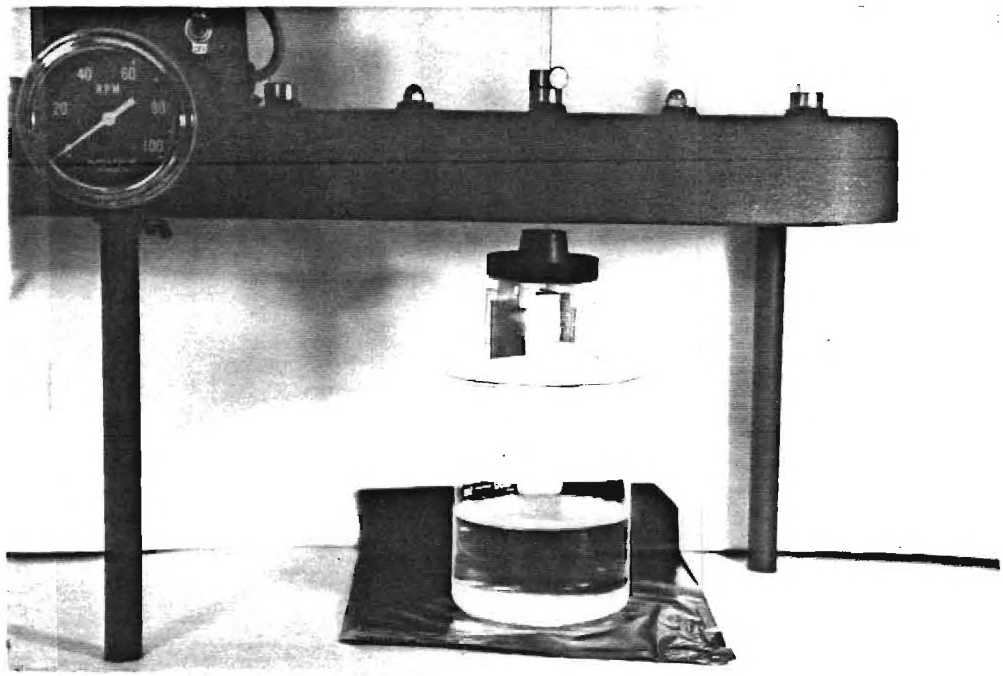
4. Coupon Adsorption Tests.

One of the topics of interest has been the identification of suitable and unsuitable structural materials for use in any lysimeter siloes that may be planned for further study of mercury migration. Preliminary experiments have been described in the previous report (2). They showed the development of a heavy scale on any galvanized iron exposed to dilute aqueous mercury solutions and helped refine the procedure used for testing.

The system adopted is shown in Fig. 2. Small (typically 25mm x 35mm) coupons of various construction materials were placed in a beaker containing dilute mercury. Three materials were chosen for the initial test; aluminum, fiberglass, and galvanized iron; all samples were run in duplicate. To remove surface contaminants that may affect mercury adsorption, the coupons were washed in a detergent solution, rinsed afterwards in deionized water and then acetone, and allowed to air-dry. The edges of the fiberglass coupons were dipped in melted paraffin to seal the ragged edges resulting from the cutting of the coupons from a larger sheet. It was felt that the edges could adsorb water by capillary forces, increasing the mercury concentration preferentially, compared with the relatively nonporous surfaces.

The dilute mercury solution was produced by placing a small drop of activated (Hg-203) mercury at the bottom of a beaker containing deionized water. After an extended period of standing the water was found to contain ~ 60 ppb of mercury. This solution was then used to measure mercury sorption on the sample coupons by counting the ^{203}Hg activity in the surface layer.

The coupons were suspended in a beaker containing 500 ml of deionized



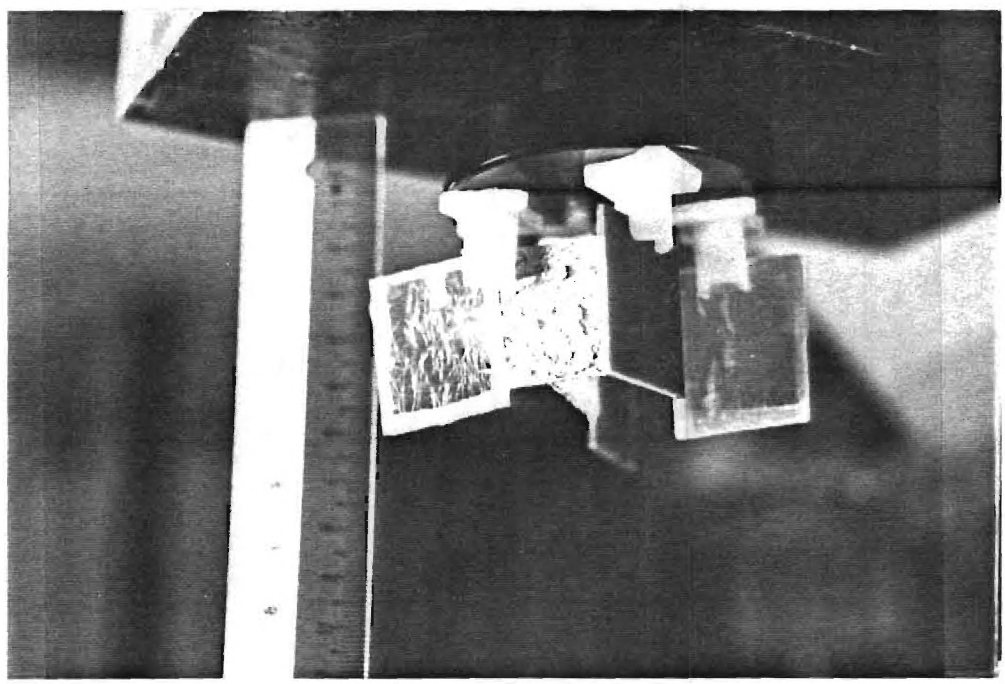


Figure 2. Coupon Adsorption Test

a. Stirrer system b. Details of coupon mounts

water in equilibrium with activated metallic mercury. The stirring apparatus was used to rotate the coupons slowly (approximately 10 rpm) to minimize concentration variations in the solution and allowed to air dry. The galvanized coupons reacted dramatically, becoming textured with a white substance while releasing a large volume of white powder that settled on the bottom of the beaker, as well as attaching to the surfaces of the other coupons. Figure 3 shows the accumulation, at 1.8x magnification. This white powder was shown to contain 0.03 % wt mercury.

To calibrate the system a standard coupon was fabricated by evaporating 1.09 g of water containing 60 ppb Hg uniformly on the surfaces of a copper coupon. This standard coupon was counted in the same manner as the test coupon and a count rate to amount-of-mercury relationship was established.

Previous tests had shown the importance of avoiding any stirring up of the mercury, which was liable to form a flocculant suspension, and of any agitation that would introduce air into the water. The sample coupons were held in teflon clips which were unaffected by the presence of mercury. In early tests duplicate coupons of fiberglass, aluminum and galvanized iron were submerged in a solution containing approximately 30 ppb activated mercury (first batch) dissolved in deionized water. The coupons were rotated at 10 rpm for a period of 3 1/2 days, at which time they were removed from solution, rinsed with deionized water, and air-dried. The coupons were counted individually using a 5-inch NaI well crystal housed in a 6 inch iron shield. The counting system used a single-channel analyzer set to accept the 0.279 MeV gamma emitted by Hg-203. The fiberglass was common sheet material of "regular" composition. The "glass" samples were microscope slides, with a polished surface, and assumed to be borosilicate

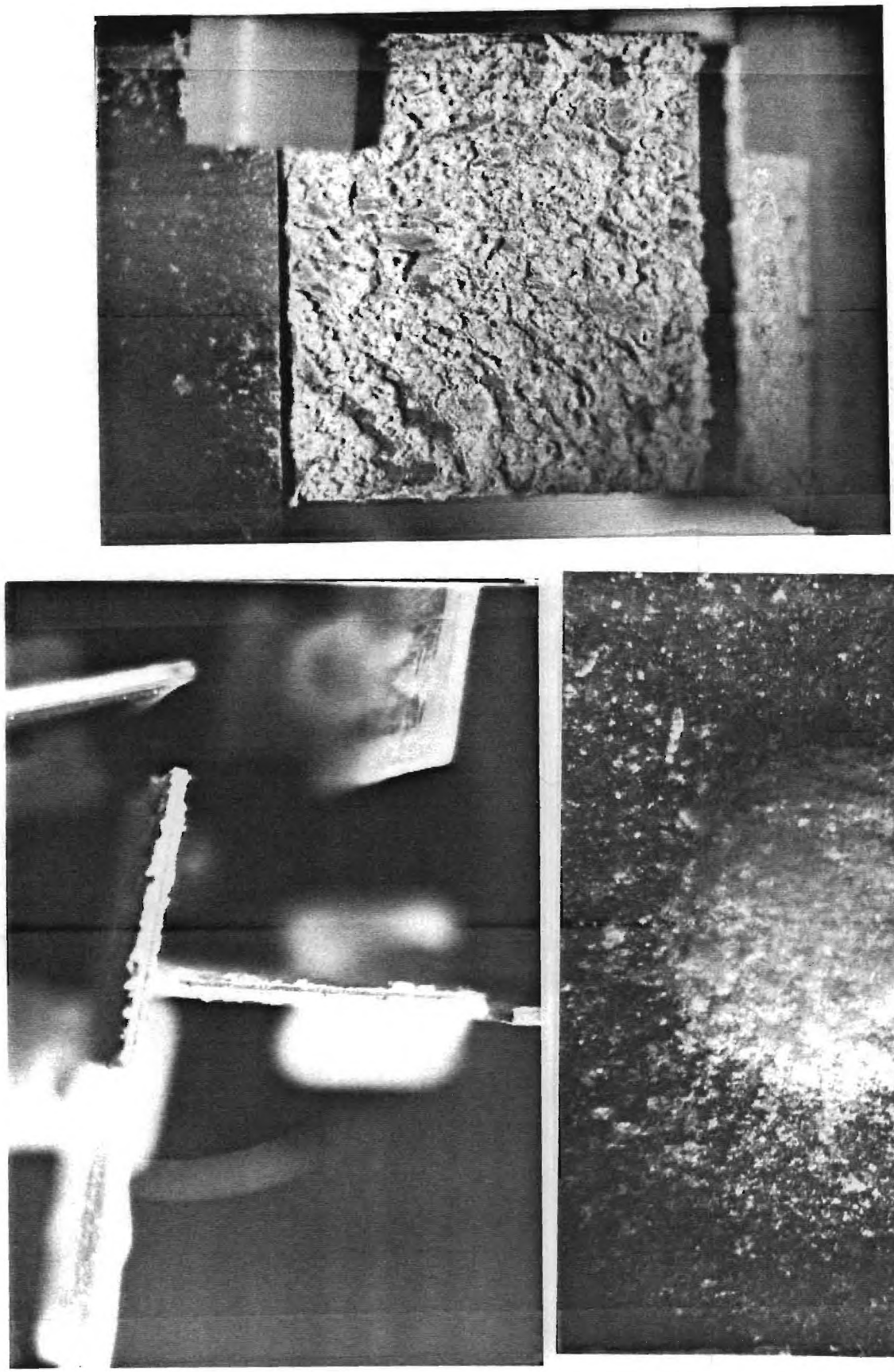


Figure 3. Dissolved mercury attack on galvanized iron.

a. View of coupon surface. b. Edge view. c. Powder in beaker bottom.

glass. Table 5 shows the results of the mercury analysis of the coupons. After counting, the coupons were rinsed with deionized water to remove the white powder and to estimate the bonding strength of the mercury. The aluminum and fiberglass coupons showed no significant reduction in activity upon washing. The galvanized coupon showed a 26.1% reduction in attached mercury.

TABLE 5

Concentration of Mercury on Coupon Surfaces (First run)

Hg Conc. (ppb)	Coupon material	Adsorbed Mercury (g/cm^2)
70	Aluminum	6.1×10^{-8}
	Fiberglass	5.9×10^{-8}
	Galvanized	3.0×10^{-7}
	Galvanized, rinsed	2.0×10^{-7}
30	fiberglass	5.3×10^{-8}
	galvanized	5.3×10^{-8} (not counting loose scale)
	aluminum	7.2×10^{-8}

The 30 ppb fiberglass and aluminum coupons compared better with the full strength test than did the galvanized coupons, which had 1/4 as much attached mercury as the galvanized used in the full-strength test. The white powder was not as prevalent as in the full-strength test. The results did not show any significant variation and it was concluded that the adsorbed activity simply had reached equilibrum with the mercury in solution. To test the time dependence a separate series of tests were run. The effect of contact time on the attachment of mercury on various construction materials was studied by placing coupons in beakers of deionized water equilibrated with mercury (approx. 60 ppb). The coupons were removed at various times, rinsed, and counted. The results are presented in Table 6.

TABLE 6

Hg Adsorption as a function of contact time

Material	Contact time (days)	Mercury concentr. (g/cm^2)
Fiberglass	1	5.1×10^{-8}
	2	4.4
	3	6.4
	10	5.9
Aluminum	1	0.12
	3.5	3.7
	10	6.2

Evidently equilibrium was reached rapidly for the fiberglass, but more slowly for the aluminum coupons.

The interaction with the galvanized iron was most dramatic and is illustrated in Fig. 3. Note that the iron surfaces were never in contact with the metallic mercury. All the effects shown were due purely to the dissolved mercury in the water at a concentration of about 70 ppb. Figure 4 shows similar pictures for the aluminum coupon and a waxed fiberglass sample. In the latter case the bright lines appear to represent light reflection off fibers in the surface layer.

A second set of experiments were conducted in November, 1985 with the re-activated tracer. In this case 25 x 35mm coupons of glass, fiberglass, Plexiglass and aluminum were exposed for 0.5 - 3 days in groups. Background counts before and after, for a 12 hr. count, were 12,100 and 12,400 counts per hour respectively. The results are presented in Table 7. Errors are shown as 2σ.

Compared with a background count rate of $12{,}250 \pm 110$ counts per hour, the net counts tabulated barely differ from zero to any degree of statistical significance. All of them appear to show a small, but finite adsorption slightly above the equilibrium concentration. This is in line with the well-known attachment of mercury to clean laboratory glassware; there does not seem to be a really significant observable difference between the samples tested, even though the fiberglass adsorption appeared to be fractionally higher than the others.

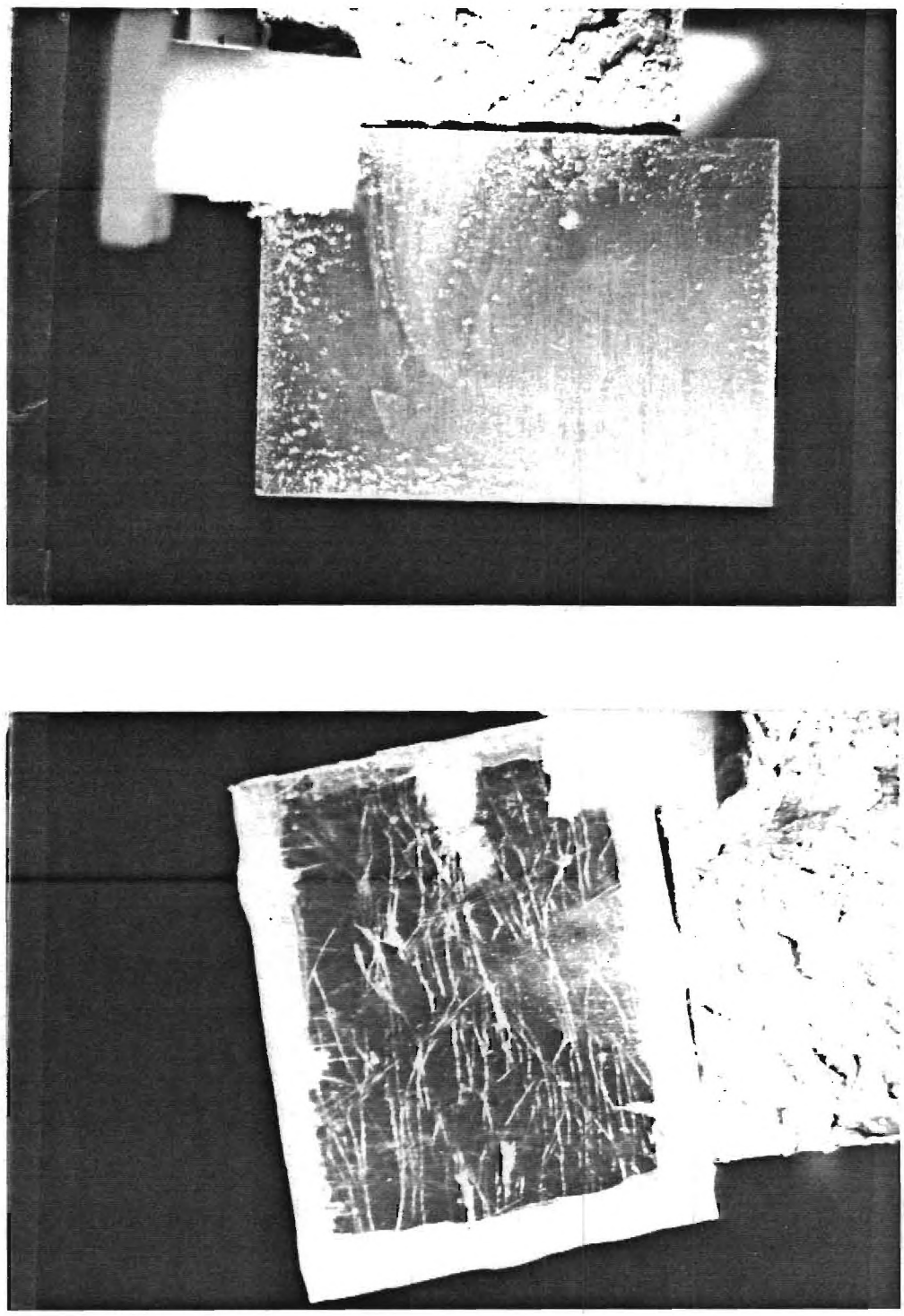


Figure 4. View of exposed aluminum (top) and Fiberglass (bottom) coupons

TABLE 7

Coupon Adsorption Tests

Material	Contact time (days)	Counting time (hr)	Net count rate (cph)
Plexiglass	0.5	2	107 ± 168
	1		25 ± 178
	2		302 ± 154
	3		605 ± 162
Glass	0.5	2	103 ± 162
	1		202 ± 174
	2		236 ± 166
	3		545 ± 170
Fiberglass	0.5	5	597 ± 158
	1		107 ± 170
	2		591 ± 176
	3		735 ± 190
Aluminum	0.5	5	355 ± 190
	1		439 ± 180
	2		431 ± 180
	3		242 ± 184

5. Seepage Column Tests

The simplest mode of migration of liquid mercury through soil would appear to be by gravitational movement through soil pores under the pressure head generated by the standing mercury and/or water column, subject to control by surface tension effects. To obtain some idea of the magnitude of this seepage process, a series of wet and dry soil columns were set up, using sand and SRP soil columns.

Orebaugh and Hale (1) have shown that in most cases such movement probably would entail volatile mercury vapor diffusion with subsequent condensation, a slow and inefficient process at subsurface temperatures. Turner (12) has done some simple calculations to relate pore entry radii with mercury pressure. For pore radii below 10 microns entry will require pressures in excess of 20 cm of mercury (column height); this assumes surface tension values of nominally pure mercury.

For experimental verification a number of packed columns were prepared in 3cm diam. glass tubes, using different sand and soil samples that had been characterized for the Lysimeter project. The surfaces of the columns were carefully leveled and smoothed. About 1 mm of clean mercury was carefully deposited and left standing. These columns were set up in Summer 1984 in a fume hood as shown in Fig. 5, and left undisturbed for over a year.

Table 8 lists the condition of the various columns. "Wet" soil was prepared by placing 50g of sand or soil in a beaker, covering it with water, and draining the excess. After long periods of standing it was assumed that each wet column contains only the appropriate minimum residual moisture concentration. The tubes were open at the bottom and loosely covered to keep out dust, but avoid any suction effects.

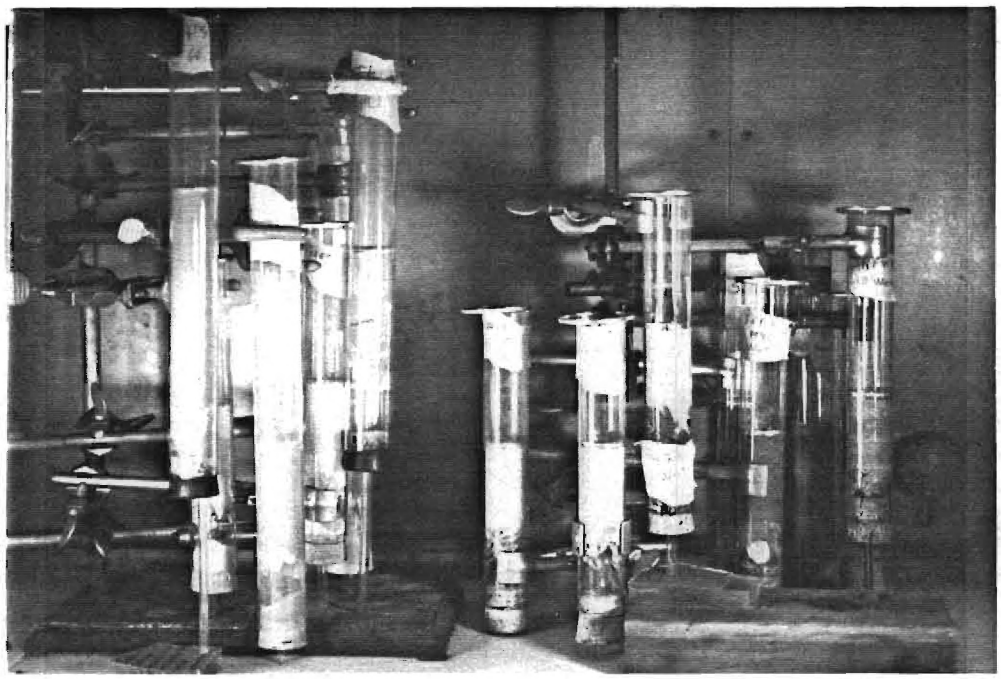


Figure 5. Set-up of seepage columns.

TABLE 8. Details on Seepage Columns

Type	SIZE	CONDITION	AMOUNT OF MERCURY ON TOP OF SOIL
SRP #1	20	Dry	41.829 g
SRP #1	40	Dry	41.774 g
SRP #1	60	DRY	42.210 g
SRP #2	20	DRY	43.109 g
SRP #2	40	DRY	44.386 g
SRP #2	60	DRY	44.710 g
SAND	20	DRY	44.044 g
SAND	40	DRY	43.668 g
SAND	60	DRY	44.050 g
SRP #1	20	WET	43.983 g
SRP #1	40	WET	43.243 g
SRP #1	60	WET	42.968 g
SRP #2	20	WET	43.861 g
SRP #2	40	WET	41.356 g
SRP #2	60	WET	43.682 g
SAND	20	WET	40.282 g
SAND	40	WET	41.959 g
SAND	60	WET	42.321 g

Since the mercury used was not radioactive, concentration profiles had to be obtained chemically. This involved extruding and sectioning the soil columns, a process that turned out to be more time-consuming and complicated than expected. In view of the low Hg concentrations expected in the lower horizons, it was very important to avoid cross contamination during the removal of both the bonded mercury and the soil columns from the glass tubes.

Column dissection procedure

a. Objective

The objective of this phase of the project was to remove the approx. 2-3 ml of standing mercury from the top of 17 soil columns and then dismantle the columns in such a way as to keep increasing by deeper strata intact for future assay.

b. Extrusion Apparatus

It was decided the best way to accomplish this was to extrude the entire column from the plexiglass tube supporting it. An extrusion apparatus was constructed for this purpose, consisting of a small steel piston head (45mm x 26 mm dia) attached to a screw threaded piston rod (300mm long). The column to be dissected was clamped to a ring stand. The nuts supporting the piston rod were clamped beneath the column and the piston was pushed slowly through the column by turning the piston rod through the stationary nuts. The transfer of the piston rod was 2 mm/revolution.

Since a tight fitting tolerance was needed, a cork stopper was placed between the steel piston and the soil surface.

The stopper was wrapped in numerous layers of clear plastic to complete the tight fit and to present an impermeable, inert surface to the soil.

c. Contamination Prevention

It was expected that decreasing concentrations of Hg would be seen as one progressed deeper into the column. As a result, precautions were taken to prevent cross contamination from one sample to the next. The more heavily contaminated strata (closest to the surface) might deposit amounts of Hg on the inside wall of the column; the wall transferring it to subsequent strata as they pass. To prevent this, the columns were inverted and extruded bottom first.

d. Dissection Procedure

1) After the piston was assembled in the top of the tube, the tube was inverted and any support materials at the bottom of the tube removed.

2) The column was then extruded at a constant rate until the bottom of the column was even with the edge of the tube. Note: only negligible column compression occurred during this step. The largest amount occurred on the longest column: ~ 1.33%.

3) As each stratum was pushed out of the tube, it was collected by scraping it off onto an 18 cm dia. filter paper with a stainless steel knife and then pouring it into a separately labeled tri-pour beaker. A fresh filter paper was used for each column and the knife was washed between columns.

4) Samples were taken every 5mm until a depth of 20 mm was reached. The top 20 mm were removed in 2mm segments.

Notes: 1. Photographs were taken of all columns prior to dissection.

2. Five columns (which were wet) were discarded as large fissures appeared when the soil dried, giving rise to uncontrolled Hg penetration. This situation is unlikely to occur at the bottom of disposal trenches.

An initial test was performed to determine the feasibility of the "screw extrusion" technique for removal of thin (1mm) layers of soil material without disturbing the remaining column. This initial test involved placing activated metallic mercury on the surface of a column containing SRP #2 soil, and subsequent removal of the ponded mercury. The column was then sectioned and each layer was analyzed for mercury concentration. The results indicated that the mercury did not cling substantially to the sides and was not smeared into the other layers as the soil column was extruded.

The column used for the migration studies was also sectioned using the screw extrusion technique. The mercury distribution with depth was compared to the distribution determined using the collimated sodium iodide crystal. This column was used as a trial run for the extraction technique in preparation for the disassembly of the static mercury columns.

The trial proceeded smoothly and the first of the static columns was also sectioned. All the columns were photographed for record purposes, sectioned, and analyzed, except for those showing visible Hg patches on the glass walls or in surface fissures.

Preparation for AA Analysis

Since the mercury was clinging to sand and soil surfaces in the columns, each soil slice had to be digested to obtain an aqueous mercury solution. The method used was EPA Method 245.5 for the determination of mercury in sediment (13).

This procedure is applicable to measure total mercury (organic and inorganic) in soils, sediment, bottom deposits and sludge type materials.

The range of this method is typically on the order of 0.2 to 5 g/g. This range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

Reagents

1. Aqua Regia: Prepared immediately before use by carefully adding 3 volumes of concentraded HCl to one volume of concentrated HNO_3.

2 Sodium chloride-Hydroxylamine Sulfate Solution: Dissolvve 12g of sodium chloride and 12 grams of hydroxylamine sulfate in distilled water and dilute to 100 ml.

3. Potassium permanganate: 5% solution, W/V. Disolve 5 grams of potassium permanganate in 100 grams of distilled water.

Procedure:

1. Triplicate portions of 0.2 grams of dry sample were weighed (totaling 0.6 gram) and placed in the bottom of an Erlenmeyer flask. Five ml of distilled water and 5 ml of aqua regia were added. Approximately 12 samples were done at a time. These were then heated at 95°C in a water bath for 2 minutes.

2. The flasks were then cooled for approximately 15 minutes and 50 ml of distilled water was added. Fifteen (15) ml of potassium permanganate solution was also added to each sample. The flasks were mixed thoroughly by hand and then placed in a water bath (at 95°C) for 30 minutes.

3. After this second heating the flasks were cooled again for approximately 30 minutes and then 6 ml of sodium chloride-hydroxylamine sulfate was added to the solution to reduce the excess permanganate and return the solution to a clear color. Another 55 ml of distilled water was added to each flask.

4. These flasks were set out to separate the sediment from the liquid for a period of about 24-48 hours, with a watch glass placed on top of the flask to prevent particles or contaminants from entering the flask.

5. These were then pipeted and transferred to Chemistry for analysis.

Selection of Material

Due to the large number of columns and "slices" from each column (approximately 180), every third one was sampled for analysis. If there was visible mercury on the column slice it was marked as "Visible Mercury", and was not sampled further.

Results

Table 9 presents the seepage results obtained for all the columns that were analyzed.

The soil samples (nominally 0.60 grams) were digested in acid and diluted to a final volume of 136 ml. The AA determines concentration in ppb. ($10g^{-9}$Hg/ml liquid). The concentration of mercury ($\mu g/g$) in a soil sample was calcutated by:

$$\frac{\text{ppb reading} \times 136}{(\text{sample wt}) \times 1000} = \mu g \text{ Hg/g soil sample}$$

Three samples containing no added mercury were analyzed along with the column samples:

Rollo sand (30-35 mesh)	6.3 $\mu g/g$
SRP 1	11.2
SRP 3	23.1

The average for the SRP soils (17.2 ± 8.4) was used as a blank correction. Reported values are analytically determined values less this blank. Rollo sand was not used because it is not typical at the soils used in this study.

All column samples were analyzed from the bottom up until mercury was found. Not all slices were analyzed, to conserve effort and to avoid too many null runs. For that reason Table 9 contains results typically only for alternate slices.

TABLE 9

Seepage Column Results

Sand	Depth (mm)	Mercury concentrations (µg/g) #20 Wet	#40 Wet	#60 Wet	#20 dry	#60 dry
	0 - 2			2993		VM (12 mg/g)
	2 - 4	VM			0	VM
	4 - 6		86.8	28.3	23.9	VM
	8 -10	86		37.8		373
	10 -12		55.2			
	14 -16				21.2	341
	16 -18		45.6			
	18 -20			0		
	20 -25	33.5			5.2	4.4
	25 -30		38.7			
	35 -40	4.9		0	0	0
	40 -45		10.2			
	45 -50					

* VM = Visible Mercury

SRP Soil

Depth (mm)	Mercury Concentrations (µg/g) #1-20 dry	#2-20 dry	#2-40 dry	#2-60 dry
0 - 2	3183			(13 mg/g)
2 - 4				
4 - 6		430	111	
6 - 8				529
8 -10	430			
10-12		365	86	
12-14				445
16-18	421	365	95	
18-20				384
20-25				
25-30		16.2	106	
30-35	21.4			6.1
35-40				
40-45		0	93	
45-50	0	0		2.6

In the coarser soils perfect smoothness of the surface is not practicable and the ponded mercury easily invaded the top 2mm. In removing the ponded mercury first, care was taken not to disturb the top layers. However, for analytical purposes that top layer usually had to be disregarded in obtaining soil column profiles.

Of the seventeen columns originally set up, five were found to have cracks that allowed gross movement of the metallic mercury. The five columns were:

SRP1 #40 wet, SRP 1 #60 wet, SRP 2 #20 wet, SRP2 #40 wet, and SRP2 #60 wet.

Although all five columns showed desiccation cracks, their appearances were unique:

SRP1 #40 wet; this column had one radial crack extending accross the top surface, penetrating to a depth of 45 mm. The crack was approximately 0.5 mm wide at the surface. Mercury was visible along the entire crack as well as in small pools (3 mm diam) adjacent to the crack.

SRP 1 #60 wet; showed the same general form as SRP1 #40 wet, except the crack width was smaller and there were three radial cracks at the surface. The cracks extended 40 mm below the surface.

SRP 2 #20 wet; the SRP #20 wet soil did not show significant desiccation cracking. Mercury was visible, however, to the 10 mm level. The mercury appeared as nearly circular droplets spread uniformly through the soil layer.

SRP 2 #40 wet; showed the same pattern as SRP 2 #20 wet, with mercury visible to 8mm. The droplets were smaller than those found in SRP 2 #20 wet, typically being up to 1mm x 2mm (width x height).

SRP2 #60 wet; this sample showed massive damage from desiccation. One half of the column had slumped relative to the other, and mercury had been released through 80mm of column. The column showed a network at large cracks, that were filled with mercury, forming reservoirs at the 50mm level.

Column samples #1-40 and 1-S-60 were lost when the AA system turned out to be either contaminated or acting erratically on the day these samples were run.

In summary, it appears that the mercury did penetrate several centimeters from the surface in both wet and dry sand columns, and somewhat more so in the dry soil. The concentrations were low, in the ppm range, and not particularly dependent on pore size. Soil cracking, as the surfaces of the initially wet SRP soil columns dried out, led to mercury invasion via the cracks in visible amounts; this conditions is not expected to arise in disposal trenches.

6. Flow Tests

Considering the relative impermeability of SRP soil, no substantial water flow is expected to occur vertically below the disposal trenches. Nevertheless, it was felt to be important to ascertain the degree of entrainment and migration of mercury that might occur under dynamic flow conditions. A series of column tests were conducted, under saturated flow conditions, to observe any movement of mercury from a localized source for a variety of flow parameters and types of fluid, using radioactive mercury tracers. The activity of the effluent was monitored, and in some cases also the profile of activity retained in the column.

Sand column tests

A 1 1/2 inch ID plastic column was packed with GT Sand to a height of 18 cm and placed in water to saturate. The column volume was 205 cm^3, the pore volume 98.5 cm^3. The column was placed in a system to allow saturated flow, with recycling, at a controlled flow rate. 0.076g of activated mercury was placed on the soil surface (6.6 mg/cm^3).

The flow rate of water through the column was controlled by a valve located at the bottom of the column. An overflow line was attached to the column above the soil level to ensure continued saturation. The water was pumped from a reservoir to the top of the column. The column effluent was collected in a graduated cylinder to verify the cumulative water flow. Samples were periodically collected from the effluent and analyzed for mercury concentration. The volume of water collected in the graduated cylinder was recorded and the water was returned to the reservoir to be recycled.

The samples were analyzed for mercury concentration using a 5-inch sodium iodide well detector housed in a 6 inch iron shield, coupled to a single-channel analyzer set to accept the 0.279 MeV photopeak from Hg-203. The minimum detectable concentration of this system for mercury in water was 20 ppb.

Effluent samples were taken at 1, 2, 4, 8, 16, 30, 40, 50, and 60 volumes and analyzed for mercury activity. The activity in all cases was below 20 ppb. The test was stopped after 60 pore volumes (6 liters) due to equipment malfunction. It was felt that the reliability of the data produced after restart did not warrant extended testing. The samples were also analyzed using Atomic Absorption to verify the results obtained from the sodium iodide system.

Vertical Distribution in the Flow Column

A sodium iodide crystal was used in conjuction with an MCA to determine the distribution of the activated mercury as a function of time and horizontal location. The detector was placed inside a lead collimator to allow high resolution during the vertical scans. The collimator had a horizontal window 3 mm high by 45 mm deep. The window gave a total scanning height at the column center of 5.6 mm. Measurements were taken at 0.5 cm intervals down the column starting at the surface. Scans were taken at three times; before start of water flow, after 30 column volumes had passed through the system, and after 60 columns had passed. 30 column volumes represent approximately 263 cm of infiltrating water, 60 volumes is approximately 526 cm, or 207 inches. Figure 6 shows the distribution of Hg activity with depth for the three scans. The mercury is seen to be moving down the column with the flow of water. After 60 column volumes (approximately 6 liters) have moved through the system, the activity at the 0.5 cm level is 24% of the activity at the column surface. The actual concentration of mercury in each horizon was not determined, initially.

Soil Column Tests

Four columns containing SRP #3 soil were set up in a once-through flow system. The columns were 28 mm diameter plastic tubes. Each column was uniformly packed to a height of 50 mm above a sand drain. The columns were saturated by submerging them in a beaker of SRP#3 equilibrated water for four days, followed by two days of flowing SRP#3 equilibrated water through the columns. The pumps were stopped and the water was allowed to drain through the column until the water level nearly reached the surface of the soil. Two drops of activated mercury were placed at the top of each

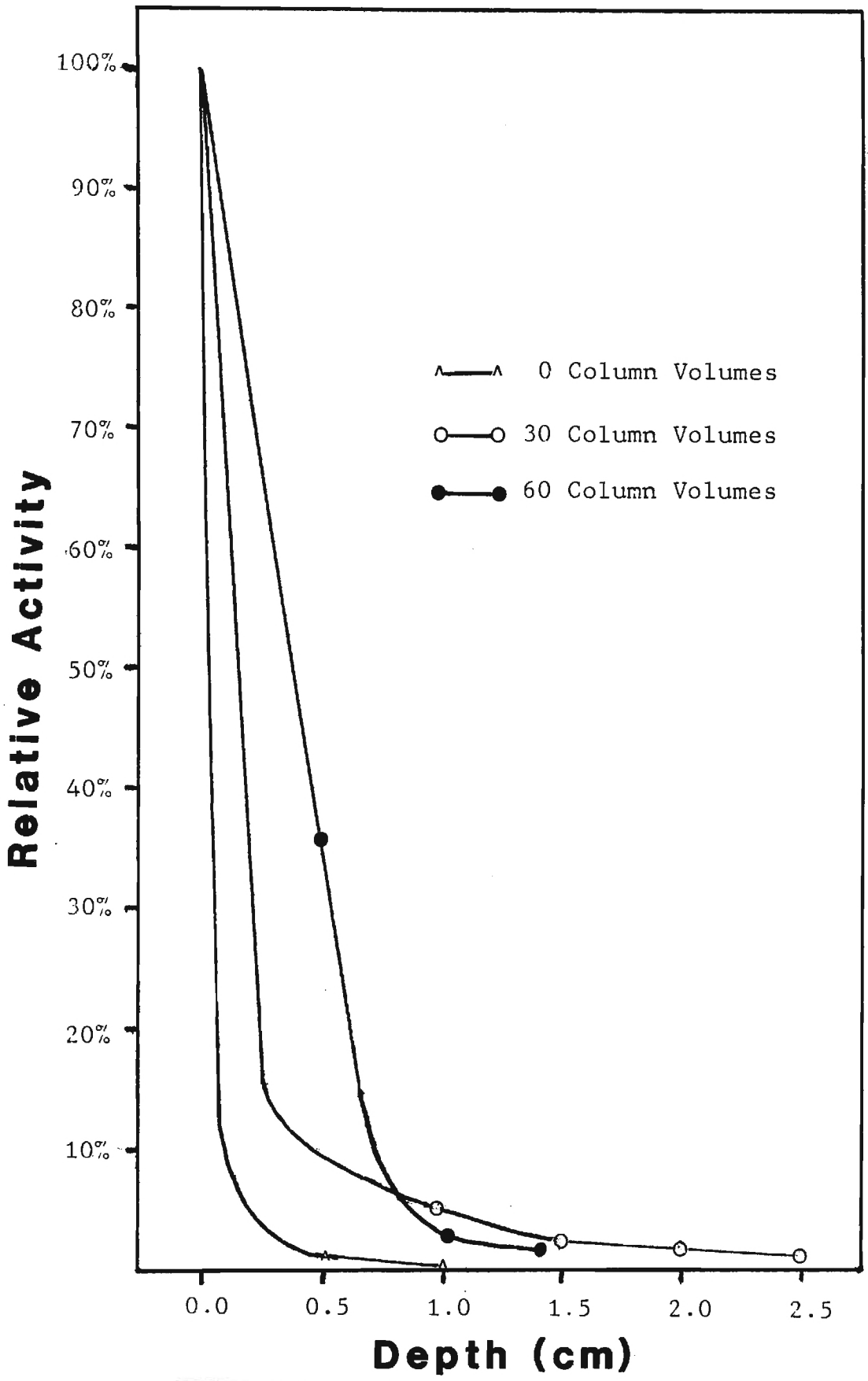


Figure 6. Mercury Profiles in Soil Columns.

column (0.170g Hg total). The drops were then covered with an additional 1 cm of SRP#3 soil. The columns were connected to their water reservoirs, which contained: Deionized water, SRP#3 equilibrated water, 10^{-4}M EDTA, and a kaolin suspension, respectively. The pumps were started and the effluent was collected after 500 ml and 1000 ml had passed through the columns. The test was stopped at 1000 ml due to the reduced flow rate through the equilibrated water and kaolin suspension columns. The effluent samples showed no significant activity. Fig. 7 shows the arrangement of the columns and the flow system.

Column Description

Diameter (inside)	28 mm
Soil Column length	50 mm
Soil Type	SRP #3
Porosity	0.48
Soil volume	307.8 cm^3
Void volume	147.8 cm^3
Flow rates	0.3-0.06 ml/min
Mercury loading	~0.04 g Hg/column (0.007 g/cm^2)

Vertical profiles were obtained using the same collimated detector as described above. The collimator was "calibrated" by packing a test column with soil and emplacing a mercury droplet to obtain initial count distributions. All counts were corrected for decay.

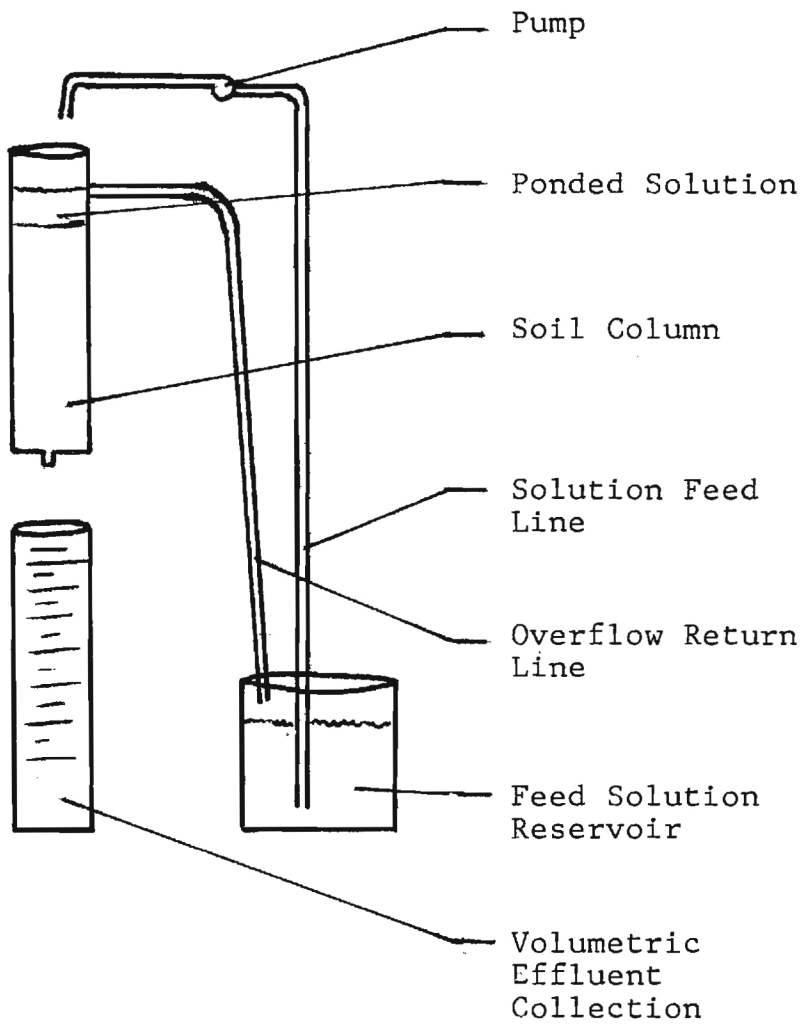


Figure 7. Experimental arrangements for dynamic flow tests.

Four columns with separate feed waters were run in parallel to maintain comparable inflow. Since the pump rate exceeded the column flow rate, the ponded water at the top of each column was kept constant by providing a lateral drain, well above the mercury drop.

Initially, the DI water and the EDTA water columns had saturated conductivities twice those of the other two columns (0.3ml/min vs 0.15 ml/min). As time passed, all columns showed decreased conductivities. The kaolin and equilibrated columns nearly stopped up (Q = 0.06 ml/min) (19 min/ml).

To test the possible entrainment of mercury by attachment to suspended clay particulates, one of the influents was an aged kaolin suspension containing micron-sized particulates in a cloudy solution. After some period of operation the "kaolin column" showed bands of white deposit in the upper half of the column, starting 1.5 cm below the mercury drop. At the end of the run the surface of the column was vacuumed to remove a kaolin layer that had collected there; about 2mm were removed. After that treatment the flow rate was 0.08 ml/min. Evidently the soil column filtered the kaolin suspension which yet passed freely through sand columns.

Results of flow tests

Table 10 presents the results of counts on the various effluent samples collected. These were obtained over a 3-week period, necessitating periodic background checks.

TABLE 10

Effluent Counts

Sample	Count time (hr)	Total counts	Gross count rate (cph)
Bkgd 1	12.77	157,124	12,308 ± 31
EDTA 1	2.02	24,732	12,243 ± 78
Bkgd 2	13.48	164,909	12,234 ± 30
Bkgd 3	3.75	46,445	12,385
Bkgd 4	12.37	153,647	12,421
DI water 1	1.43	18,053	12,629
DI Water 1	9.47	115,340	12,180
EDTA 1	12.05	147,924	12,276
Equil. 1	11.92	145,984	12,247
Kaol. 1	12.03	148,103	12,311
Kaol. 2	11.97	147,695	12,339
Equil. 2	12.20	146,178	11,982
EDTA 2	11.75	145,303	12,366
DI Water 2	25.45	319,134	12,540
Bkgd. 5	16.35	198,289	12,128 ± 27

It is evident that the system background was quite stable over the test period and none of the effluents showed any activity significantly different from background.

Since the mercury did not move significantly from the layer which included the droplet, profile measurements with the collimated detector were confined to a thin region just below that layer, where the count rate was highly affected by the dominant activity from the droplet, which raised the effective background through leakage through the collimator wall. The detector background itself was determined as 21±3 cpm.

The column was positioned in front of the collimator with the mercury drop in line with the window. Short counts were taken and the column was moved slightly up and down until the count rate was at a maximum. This was marked as "slice 1" and a 1 minute count was taken. The column was moved 0.5 cm and another count was taken. This process was repeated until the count rate reached the background count rate. 'Approximately 6.7 column volumes of water had passed through the system, or 162 cm of infiltration. This would represent 64" of rain (assuming no run-off) or 256 inches (assuming 25% infiltration). The flow rate was: 70-14 cm/d.

A "blank" run was done to determine the effect of the mercury droplet in the absence of any water movement. The results are presented in Table 11, both as gross counts and as relative activity. The distributions in all four columns were similar to the blank column. It is evident, that even after 6.7 column volumes of water had passed through the system, no mercury movement had occurred.

Table 11

Count Profiles for Flow Columns

Depth (cm)	Kaolin (cpm)	Kaolin % of max.	EDTA cpm	EDTA % of max.	DI Water cpm	DI Water % of max.	Equil. cpm	Equil. % of max.	Blank cpm	Blank % of max.
0-1	2474	100	3043		2678		2536		3678	
0.5-1.5	704	27.8	1283		1230		1134		1435	
1-2	45	1.2	58		35		36		41	
1.5-2.5	23	bkg.	17	Bkg.	23	Bkg.	20	Bkg.	21	Bkg.

CONCLUSIONS

The work described in this report had two objectives: a. to provide experimental evidence for the movement of metallic mercury through SRP soil under conditions simulating those pertaining to the bottom of a water-filled disposal trench; and, b. to provide evidence for any further test work that may be required or remedial action that may be called for to prevent mercury infiltration into local aquifers.

On the basis of the work reported here, the following conclusions may be drawn:

1. Water in prolonged contact with clean mercury metal may dissolve mercury in concentrations of the order of 30-70 ppb. This is comparable with the mercury concentration in sea water, 0.03 mg/ml (14), and well below drinking water standards, about 0.5 ppm.
2. EDTA in high concentrations will complex the mercury and result in higher Hg uptake. However, the concentrations involved seem to be higher than would be expected to occur in general reprocessing waste solutions.
3. Suspended clay minerals in water do not attract soluble Hg significantly in otherwise soil-equilibrated water and do not form an important migration pathway.
4. Standing metallic mercury may penetrate into the ground under its own head. Observed penetrations into dry soil were of the order of 3 - 4 cm. Although no time dependence was established, it is probable that the mercury would not migrate much further unless substantially higher hydrostatic pressure were applied. Mercury moved further where wet soil was allowed to dry and crack, but that condition is not expected to prevail at the bottom of a burial trench.

5. Flow tests over emplaced mercury drops with equilibrated water, dilute EDTA and kaolin-containing water all showed no demonstrable migration or entrainment.

On balance, it appears that the mercury present in the SRP disposal trenches would constitute of negligible health hazard. Some Hg dissolution, in the 30-70- ppb range, may occur under saturated flow conditions; however, normally this flow would be slow and would not significantly add to other, more important contaminants that may arise.

Supplementary tests on construction materials for the assembly of lysimeters systems have confirmed that galvanized iron is an effective collector for mercury and wholly unsuitable in contact with even highly dilute mercury-bearing water. All of the other materials tested appear to be equally suitable for this purpose in this respect.

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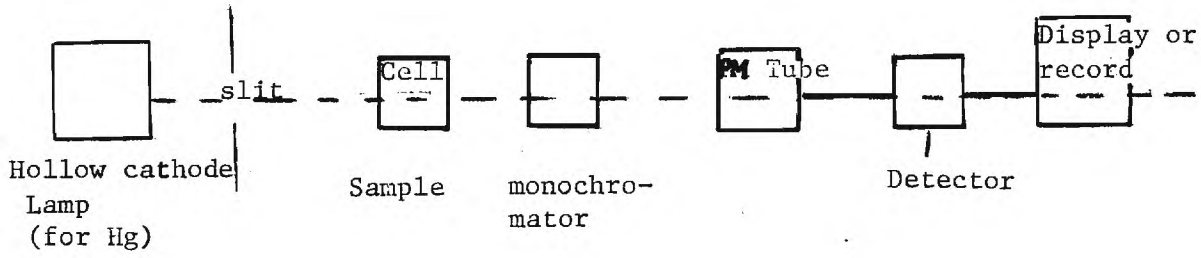
APPENDIX A

Atomic Absorption Spectrometry

In this method we use a lamp with a hollow cathode made with the element of interest (in our case with mercury, Hg). The cathode is heated and the specific emission occurs: the light emittted is characteristic for a given element (to avoid any absorption by other elements present in a sample) - it is a spectrum consisting of lines characteristic for atomic (not ionic) energy levels of a given element.

AAS METHOD



The light emitted by the cathode passes through a slit and gets into a cell with a sample being carried through. If there is any mercury in the sample, the specific absorption occurs: the light of the characteristic spectrum is absorbed by our element. This causes a decrease in the intensity of the light transmitted and the signal can be recorded. The decrease should be proportional to the amount or concentration of a given element.

A monochromator serves to choose the best spectral line for the element: lines have different intensity and so can be used for various concentrations of the element (less sensitive or less intense lines for larger amounts of the element).

The signal for a wavelength chosen by the monochromator needs then to be amplified by a photomultiplier tube and transformed to an electric current (same device) which can be then measured by some electric devices and compared

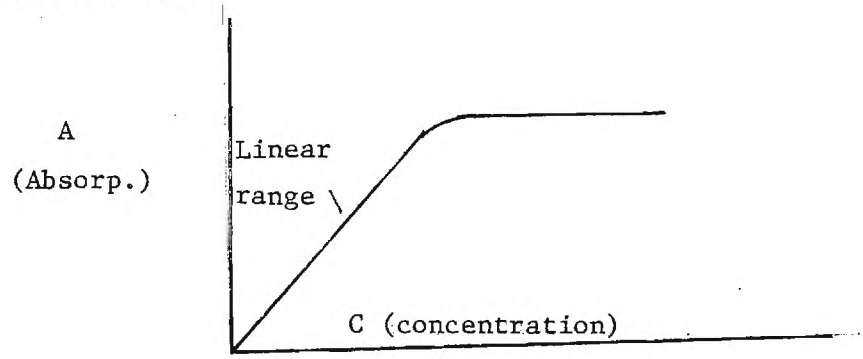
with the signal for "background" (no sample in the cell) to get the difference proportional to the concentration of the element. This signal can then be displayed or recorded.

Important things to know about:

1. The peak height may or may not be proportional to the concentration of the element (it often is so for very sharp peaks).

2. The peak area always is proportional to the amount or concentration of the element. It is always safer to use peak area to compare sample with standards.

3. The relationship absorption vs. concentration of the element (for a given volume, or amount if volume changes) is linear only in a certain range of concentration. It looks like this:



for λ = 253.7 nm for Hg (mercury) the range is 0-100ppb, but the minimum detectable amount is $\sim$ 10ppb (2-3 times background).

Possible Sources Of Error:

1. If a wavelength is not set up exactly on the peak (max line intensity) -the absorption is no longer proportional to the concentration. To check this, you can scan the spectrum around λ=253.7 nm and pick up the value for which the display is max.(If the instrument is drifting -this causes serious problems and has to be checked many times during measurements.

2. If the lamp is not well focused - same problems. To check: hit set up and maximize the display, also keep checking energy (should be 60-70) while working.

3. The cell not well placed in the light pathway: light reflects from cell walls etc. To check, use other lamp (with visible light) and make sure that the light is centered in the cell.

AAS Set-up Procedure

Cell - 4.9 HGT, 5 HORIZ

Apparatus Setting Sheet.

1. Set 5500 to run (usually the instrument is "on" all the time).

2. Warm lamp, press 8 [LAMP MA] (before press 2 [LAMP #] check the #).

3. Set λ , press 253.7 [λ PEAK].

4. Hit [SETUP], get display of arbitrary No. (45-55). Maximize display (screws on the lamp), hit SETUP again. Check the energy (should be high, <60).

5. Buttons illuminated: [AA] [ABS] [REC ABS] [PEAK HGT]

6. Set zero by initiating READ cycle: Push [READ], push [AZ.] (Zero set up)

7. To read, push READ. Record peak height displayed, to read peak area,

push [PEAK AREA] record then push [PEAK AREA] again.

NOTE: Do not change "peak area" to "peak hgt" or vice versa while measurement is being done (the instrument would stop measuring and you would lose the sample introduced, with no real result).

SLITS: H 0.7: press 0.7 [SLIT HIGH]

TIME: 30s: press 30 [t]

NOTE: If anything happens with the instrument so it stops working and you can not push any button - try to press CE to clear it and then try again what you need. If it does not help - call somebody to help you.

NOTE: Before you can do anything, check if the instrument is on "AA" mode (if on "ICP " turn the knob placed where the lamps are - above and deeper than the lamps).

NOTE: Check the cell positioning using any other lamp (pink light is easier to see) at an operating current (not max). If not centered, turn knobs at the bottom of the burner.

Cold Vapor Technique Parameters:

Flow rate must be = 500m ml/min. Adjust with the valve (green top).

$SnCl_2$ to the reaction vessel: 1 ml of Sn Cl_2 before each measurement, then hit READ to find the background and after display is obtained, hit AZ to set up "0" on the background. (Automatic pipet).

Volume of sample - 1 ml (disposable syringe).